DESCRIPTION

The present invention relates to the preparation of lactams which are used as base monomers for polyamides. More specifically, it relates to a process for the preparation of lactams from the corresponding cycloalkanone oximes by rearrangement according to the Beckmann reaction, in which process methanesulphonic acid is used.

Background of the Invention

The Beckmann rearrangement reaction, which consists in converting ketoximes to the corresponding substituted amides by means of acidic reagents, has been known for a very long time.

This reaction is taken advantage of in the industrial production of lactams from cyclic ketoximes, more particularly in order to form caprolactam and lauryllactam, which are the base monomers of polyamide-6 and polyamide-12 respectively.

Provision has been made for the use of various acidic reagents in carrying out the Beckmann rearrangement.

The use of sulphuric acid, alone (see DE-B-15 45 653 and FR-A-2 417 501) or as a mixture with trifluoroacetic acid (see JP-A-51034185) or sulphur trioxide and chlorosulphonic acid (see JP-A-57031660), has been disclosed.

Provision has been made for the use of phosphoric acid (see CH-A-530402 and JP-A-62149665) or polyphosphoric acid (see DE-B-1 545 617).

The use has also been disclosed of acetic acid (see CH-A-394212), of a mixture of acetic acid and cyanuric acid (see JP-B-71023740), of a mixture of acetic acid and acetone (see JP-A-51004163), of a mixture of acetic acid, acetone and a fluorinated catalyst (see JP-A-51004164) and of a mixture of acetic acid or acetic anhydride and hydrofluoric acid (see US-A-3 609 142).

Finally, provision has been made for the use of hydrochloric acid in conjunction with a polar organic solvent (see DE-A-1620478) or with a catalyst, for example a metal salt (see US-A-3 904 608) or a mixture of silica and alumina.

Sulphuric acid is by far the most commonly employed acid on an industrial scale. However, sulphuric acid is not without disadvantages.

It is known that, under the temperature conditions of the rearrangement (greater than 135°C), sulphuric acid is a factor which promotes the appearance of hydrolysis side reactions. This hydrolysis takes place on the starting cycloalkanone oxime, which it converts to ketone, on the one hand, and on the final lactam, which it converts to amino acid, on the other hand. This results in a decrease in

The reaction is generally carried out at a temperature of between 120 and 180°C, preferably 140 and 160°C, and for a period of time such that the residence time in the reactor varies from 2 minutes to 1 hour, preferably 15 to 30 minutes.

The rearrangement is carried out with vigorous stirring. In the present invention, the expression "vigorous stirring" is understood to mean stirring which exhibits a Reynolds number (Re) of greater than 10,000, calculated according to the formula:

$$Re = l^2 n\rho/\mu$$

in which

l is the diameter of the stirring component

n is the number of revolutions per second

ρ is the density of the reaction mixture

μ is the viscosity of the reaction mixture.

On conclusion of the reaction, the lactam is recovered in the methanesulphonic acid. This solution is generally subjected to one or more separation and purification stages well known to a person skilled in the art. The recovered methanesulphonic acid can easily be purified, for example by simple distillation, in order to be able to recycle it in the process.

The examples which follow make it possible to illustrate the invention.

EXAMPLE 1

231 g of a solution comprising 31% by weight of cyclododecanone oxime (0.363 mol) in methanesulphonic acid are added over 1 hour to 100 g of 90% by weight methanesulphonic acid, which acid is maintained at 120°C with stirring (Re > 10,000). The reaction mixture is brought to 135-140°C for 1 hour in order to bring the rearrangement to completion.

At the end of the reaction, 70.9 g of lauryllactam are recovered (yield: 99%). No trace of amino acid resulting from the hydrolysis of lauryllactam is found.

EXAMPLE 2 (COMPARATIVE

250 g of a solution comprising 30% by weight of cyclododecanone oxime (0.38 mol) in sulphuric acid are added over one hour to 100 g of 98% by weight sulphuric acid, which acid is maintained at 120°C with stirring (Re > 10,000).

After reacting for 1 hour at 135-140°C, 73.12 g of lauryllactam are recovered (yield: 97.5%).

In addition, the reaction mixture comprises 1.125 g of cyclododecanone and 0.75 g of 12-aminododecanoic acid.